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# Alpha activities and mass number assignments of light astatine isotopes produced by heavy ion reactions

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With 8 figures in the text

ABSTRACT

In order to study neutron-deficient astatine isotopes, gold has been bombarded by carbon and neon ions in the Stockholm 225-cm cyclotron. Chemical isolations and mass separations have been made and the alpha activities found have been determined with respect to half-lives and energies by use of ion chamber technique. The isotopes $^{202}$At, $^{203}$At, $^{204}$At, $^{205}$At, $^{206}$At and $^{207}$At were studied in this work.

## Introduction

Cyclotron-accelerated carbon and neon ions have been used at the Nobel Institute of Physics in Stockholm for bombardments of different heavy element targets in order to produce neutron-deficient nuclides in a still heavier element region [1–9]. In a series of such bombardments started some years ago, gold was used as target material. Similar experiments had also been done by others, for instance by Hollander in Berkeley [10].

In 1956 we found some new alpha activities in the astatine fractions. It could not, however, be entirely excluded at that time that these activities were due to decaying polonium isotopes formed from astatine isotopes by electron capture decay. A thorough investigation of the alpha-decay properties of light polonium isotopes [2, 6, 7, 9] showed, however, that the activities in question had to be ascribed to astatine.

About ten years ago light astatine isotopes were studied by Barton, Ghiorso and Perlman [11] from bombardments of bismuth with high energy helium ions. Other groups have also made similar investigations [12–16]. Because of the complexity of the isotopic mixtures, half-life determinations and mass number assignments were in most cases made by the study of known decay products of lower atomic numbers. The results were considered as uncertain, which is shown by the B and D classifications of the isotopes of interest in the Berkeley table of nuclides [17]. Recently, in a conference communication, Hoff, Asaro and Perlman [18] reported new, accurate measurements, which were mainly in agreement with our results. These authors also gave good mass estimations based on genetic relationships.

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In our studies we have been able to make direct mass assignments by use of the isotope separator of our institute. A new method has been developed by which also relatively short-lived nuclides could be mass assigned [19].

## Bombarding technique

Gold foils were bombarded in the 225-cm cyclotron [20] with internal beams of $^{12}C^{4+}$ and $^{20}Ne^{6+}$ ions. General acceleration data for these heavy ions and a general description of the bombarding techniques have been presented elsewhere [21]. Both thick-target arrangements and thin-target, recoil collection technique have been used in our experiments [7, 9].

The bombardments were made at various radii. For carbon ions 87–90 cm radii were generally used, giving nominal energies of up to about 130 MeV. The intensities of the carbon ion beam reaching the target holder with energies above 90 MeV were usually in the range of $10^{-2}$–$10^{-1}$ $\mu$A. About 63 MeV were needed for the penetration of the Coulomb barrier.

For the neon ion irradiations the radii were usually about 80 cm, for which the nominal energy of the ions was about 170 MeV. Owing to the shape of the continuous energy distribution for the 6-charged ions [21], the majority of the reaction-capable particles had energies not much above the Coulomb barrier, which in this case was around 103 MeV in the laboratory system. The intensities of the neon ions reaching the targets with energies above this barrier value were in the range of $10^{-3}$–$10^{-2}$ $\mu$A.

The irradiations lasted from about ten minutes to some hours.

The probes had been designed in such a way that the foils in both types of target arrangements used could be quickly removed in order to facilitate the study of nuclides with short half-lives [21].

By the use of the recoil collection technique the nuclear reaction products, formed in the 1–10 mg/cm$^2$ thick gold targets and thrown out of these foils, were caught in 1–2 mg/cm$^2$ thick organic films. These films were made of Tygon or VYNS, a copolymer of polyvinyl chloride and polyvinyl acetate. They were mounted so that each one was placed behind a thin gold target in the stack-of-foil arrangement used [7].

## Chemical procedures

In the bombardments of gold with carbon and neon ions the nuclear reaction products and their daughter nuclides were formed within a wide range of atomic numbers. Since the astatine isotopes of interest in this study had rather short half-lives, a rapid chemical isolation procedure had to be used.

In some preliminary experiments astatine was distilled into toluene after dissolution of the thick gold targets in aqua regia. From the toluene solution X-rays were studied. The gas was in some experiments also trapped in sodium hydroxide solution. However, the yields were low.

The fact that the dissolution of the gold targets had to be made in strongly oxidizing agents was not favourable for the astatine separation. Several of the separation methods described in the chemical and physical literature and also modifications of them have been tried, as for instance:

(1) Solvent extraction by use of di-isopropyl ether (DIPE) [11].
(2) Extraction by a solution of tributyl phosphate (TBP) in butyl ether [22].
(3) Sublimation onto a "cold finger" from the melted gold target [23, 10].
(4) Ion exchange separations.

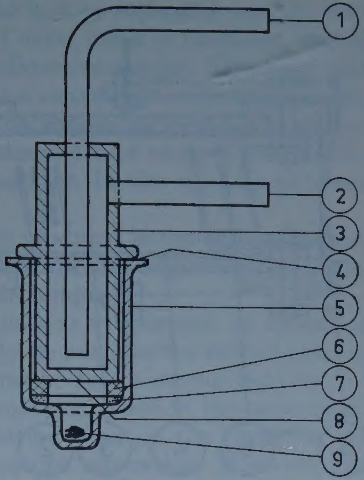

Fig. 1. Cold finger arrangement for sublimation of astatine. 1, inlet for cooling agent; 2, outlet for cooling agent; 3, stainless steel tube with collar; 4, ground surface; 5, fused silica crucible; 6, stainless steel ring; 7, asbestos ring; 8, collector foil; 9, gold target.

The apparatus is twice the size shown in the figure.

A short review of astatine separation methods is given by Bagnall [24].

Chemical separations were made only after the thick-target irradiations, where the useful nuclear reaction yields were much higher than in the recoil technique experiments. The separation procedure used in our final experiments was as follows.

Immediately after the bombardment, the approximately 100 mg/cm$^2$ thick gold targets were dropped into a transparent, fused silica crucible fitted with a stainless steel cold finger to which a platinum collector foil was clamped as shown in Fig. 1 [10]. The finger had been cooled down to almost $-80°C$ by a dry ice–acetone mixture. The gold foil was rapidly melted by strong heating of the bottom of the crucible by use of an oxy-acetylene blowpipe. After about half a minute the platinum foil was taken out of the crucible for further chemical astatine separation, isotope separation or direct counting.

The further chemical separation from possible contamination of, for instance, polonium, was made by dissolving the astatine in a few drops of concentrated hydrochloric acid to which ferrous sulphate had been added to keep the astatine in the zero valent state. After washing of the platinum foil by some further drops of this solution, an equal amount of DIPE was added. By rapid stirring of the two phases in a small tube, astatine was extracted into the organic phase, which was afterwards washed once by hydrochloric acid.

The DIPE solution was cautiously evaporated in a small platinum dish, which was finally measured in an ion chamber.

## Electromagnetic isotope separation

At our institute an electromagnetic isotope separator has been in operation since 1948 [25]. It has been used for preparation of targets of stable isotopes and for separation of radioactive material, especially products formed in neutron-induced fission. Recently a thorough reconstruction has been made [26].

In principle, the problems of mass separation of products from heavy ion reactions are, of course, not different from that of, for instance, neutron-induced fission pro-

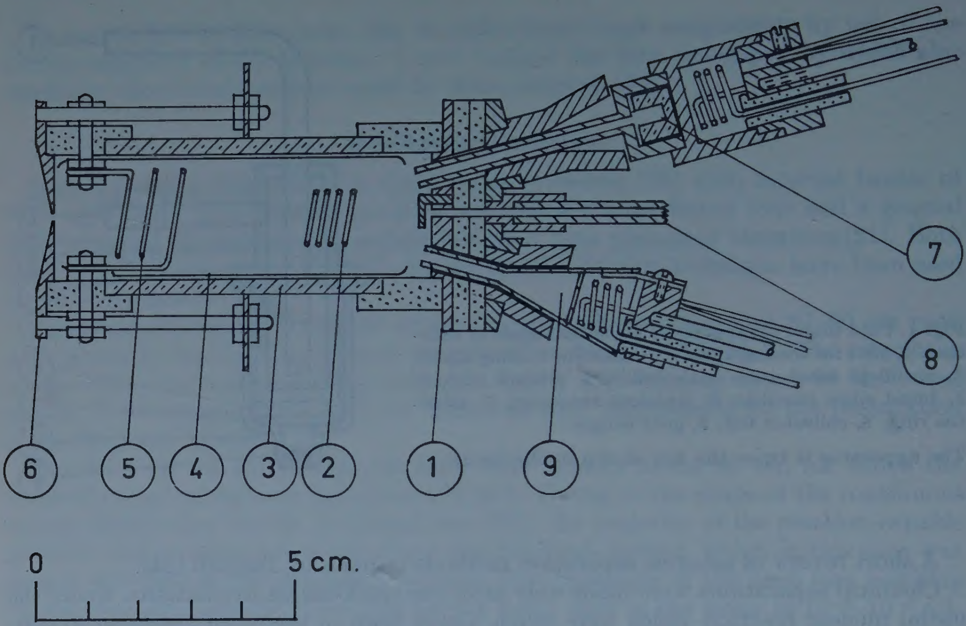

Fig. 2. Schematic drawing of the isotope separator discharge chamber seen from above. 1, back end plate; 2, cathode; 3, insulating wall; 4, anode; 5, heating filament; 6, outlet plate; 7, stable material furnace; 8, gas inlet; 9, radioactive material furnace.

ducts. The yields in the heavy ion reactions are, however, often very low, mainly due to the relatively low intensities of the cyclotron heavy ion beams. This fact places high demands on any preparation or separation procedure of the activities obtained.

In the present case, the time and the efficiency factors were of great importance. As short-lived isotopes were to be separated, it was necessary to utilize a new, rapid method [19]. The separation time was only a few minutes, which should be compared with the normal separation time of about one hour. The method used also gave a 50 % increase in efficiency, as no active material was lost in the process of achieving stable discharge conditions in the ion source. The efficiency is here defined as the ratio between the collected material and the amount put into the ion source of the separator. In the present case it was 5–10 %. The ion current in our separator, which is of the low intensity type, was of the order of 10–50 $\mu$A.

The resolving power obtainable was roughly 1000. This corresponds here to a mass distribution half-width of about 1.5 mm at a mass dispersion of 8.5 mm.

When carrier-free radioactive material or material with an amount of carrier less than some micrograms is to be isotope separated it is sometimes possible and convenient to use the special time-saving method mentioned. In the present case this method has been used for all isotope separations.

The separation procedure was, in short, the following. To the discharge chamber in the ion source of the separator two ovens could be connected as shown in Fig. 2. One of these was charged with a few milligrams of a compound, for instance, lead chloride, which has similar properties concerning the discharge conditions required etc. as the radioactive material. A quantity of about 3 mg was necessary to maintain a discharge in the ion source for about one hour. The isotope separator was put into operation with the use of this furnace. The second oven, made of platinum, was of low heat capacity. It was charged only with the radioactive sample. This oven was connected to the discharge chamber and heated at a convenient moment when the separator had reached stable running conditions, as shown by a well-focused ion beam at the collector. In fact the cyclotron bombardments were stopped when stable running conditions were obtained in the isotope separator. In this way a separation could be made within a few minutes.

As mentioned above, the astatine isotopes had been produced in two different ways, by carbon and by neon ion bombardments, and by two different target arrangements, the thick-target technique and the recoil collection technique.

After the thick-target bombardments, the astatine was evaporated, as described above, onto a thin platinum foil, which could quickly be put into the second oven of the separator. It was found, however, that the astatine had a strong tendency to leave the platinum foil too early and to give an insufficient yield in the separation. The results became somewhat better when the astatine layer on the platinum foil was covered by a thin layer of zapon before the isotope separation. However, the degassing of the zapon made the conditions in the isotope separator less stable.

More positive results were obtained with the recoil collection technique. Here, after the cyclotron bombardments, the organic catcher foils were cut out from the aluminium rings to which they had been attached. The foils were then placed directly into the platinum oven of the isotope separator, which was then quickly run. With this method no chemical isolation was made before the isotope separation. The different alpha activities obtained in the aluminium collector foils of the isotope separator could, however, be compared with the corresponding activities found after chemical separations. Thus, determinations of both atomic and mass numbers for an active nuclide were made possible.

## Counting technique

The determination of the alpha-decay properties of the various nuclides studied in this work was made by means of an ionization chamber and a 50-channel pulse-height analyzer described earlier [27]. Each mechanical register of the pulse-height analyzer has an extra contact which was used to activate a 36 channel recorder (Hasler AG, Bern) of a type ordinarily used to monitor industrial processes. This instrument recorded every alpha count with respect to energy (channel number) and time of occurrence. It thus gave a permanent record from which the alpha spectra and the decay curves could be accurately constructed. An example of such a recording is given in Fig. 3. Since the activities studied were very weak, the poor time resolution (0.1 sec) could be tolerated.

After isotope separation, samples for the alpha measurements were obtained by cutting the aluminium strip used as the catcher in the separator to pieces, in such a way that each piece corresponded to one mass number. The aluminium plates so obtained were introduced into the ion chamber and their activity was studied.

Because of the fast stopping of the low energy ions in the isotope separator [cf., e.g. ref. 28], the samples prepared in this way were very thin and were well suited

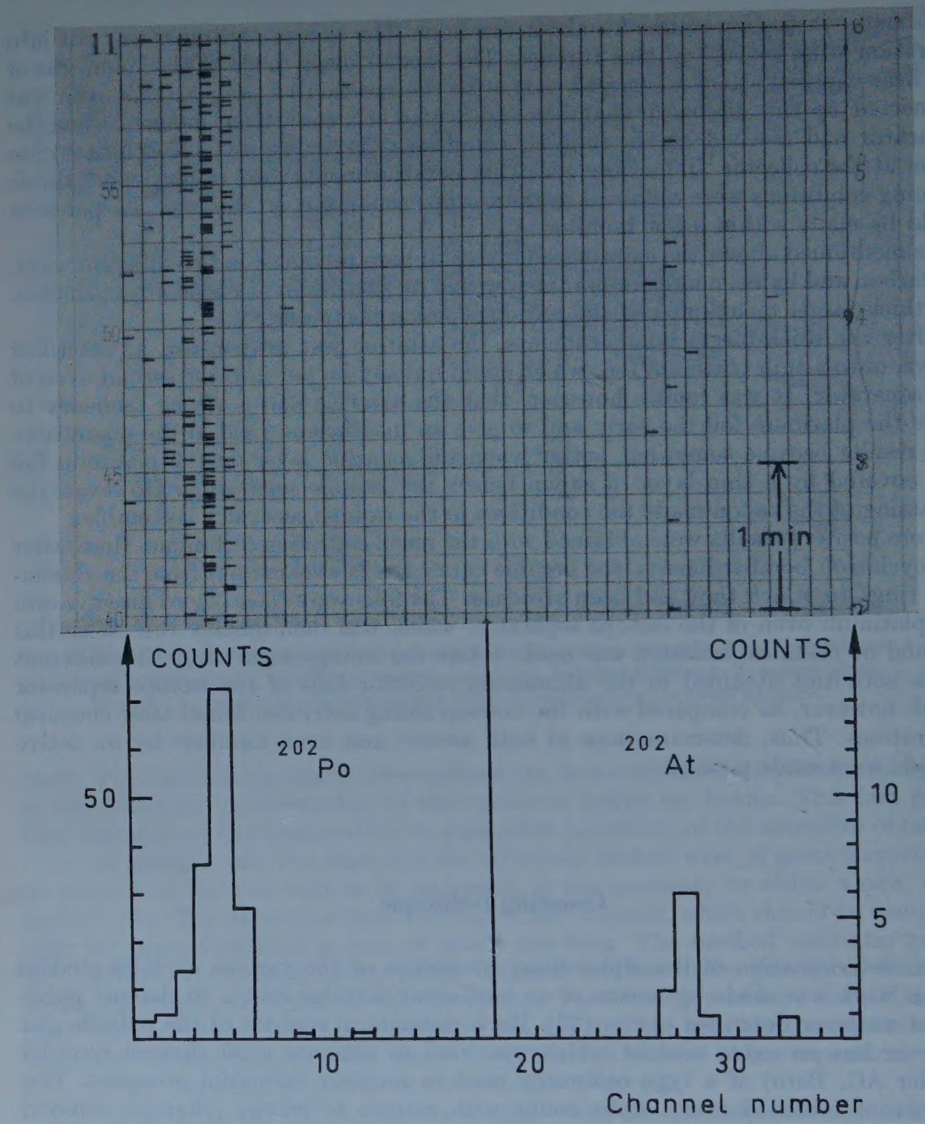

Fig. 3. Example of the recording of alpha counts and the corresponding alpha spectrum obtained from it. This record is taken from a measurement of the mass 202 spectrum.

for alpha measurements. No broadening of the alpha peaks due to absorption could be seen, as compared with a $^{210}$Po sample prepared by distillation. The full width at half maximum of the peaks in this case was about 30 keV. In the energy range studied, the background of the ion chamber measurements was less than one count in ten hours per channel.

## Results and discussion

The results obtained in our study were in some cases rather different from those reported by other authors. The two short-lived alpha activities of astatine found in this laboratory in 1956 [21] have now been identified as alpha activities from $^{204}$At and probably $^{202}$At.

Two alpha-particle energy spectra obtained from carbon ion bombardments of gold are shown in Fig. 4. In these cases no isotope separations had been made. In one of the two experiments only sublimation of the astatine from the gold target had been carried out. In the second one also the above-mentioned DIPE-chemistry procedure had been used, which to a large extent had reduced the amount of polonium in the sample, as seen from the ion chamber measurements. Further data about these runs are given in the legend of Fig. 4. It should be pointed out that our conclusions about the different activities have been based on several such spectra. The reproducibility of the activity peaks has, for example, been tested in different channel number regions to avoid possible errors arising from small differences in the channel widths. In Fig. 4 these widths have been chosen so large that the whole spectrum could be obtained on the same recorder paper. This is, of course, not always desirable, as the number of channels of one activity peak would then generally become too small.

The decay data presented below were generally those obtained after isotope separation. As the total yields of the activities were very low, the statistical basis for the disintegration data was poor. However, the results were in most cases sufficient for the identification of the activities.

The measurement data have been treated in the following way. The number of counts obtained in the different channels of the pulse-height analyzer were plotted against the mean energy values of the channels as shown in Fig. 5 for the mass 206. In this case three spectra were taken, two from bombardments of gold by neon ions, I and II, and one from a bombardment by carbon ions, III. The sum of the spectra has then been used in Fig. 7, where the counts have been plotted into energy intervals of 0.02 MeV. The counts from, for instance, Fig. 5, which were on the border-lines between these energy intervals in Fig. 7, have been divided equally between adjacent units.

The results of the experiments for masses 202–204 are shown in Fig. 6 and those of the masses 205–207 in Fig. 7. A detailed discussion will be given below for each mass number. The results are based on altogether six bombardments with carbon ions and six with neon ions.

### *The nuclides* $^{<202}$At

No alpha activities of $^{201}$At or still lighter isotopes were found after isotope separation or full chemical isolation because of their short half-lives compared with the separation time needed. However, after only rapid sublimation, it was possible in the neon experiments to find the short-lived alpha activities with the particle energies, $E_{\alpha p}$, close to 6.35 MeV and 6.50 MeV which had earlier been reported [11]. In both cases the spectra seem to indicate the presence of fine structure.

### *The nuclide* $^{202}$At

In a few of our experiments it was possible to start the alpha measurements soon enough after the bombardments to be able to register data from the decay of $^{202}$At. The identification of the alpha activity was made in different runs, including sublima-

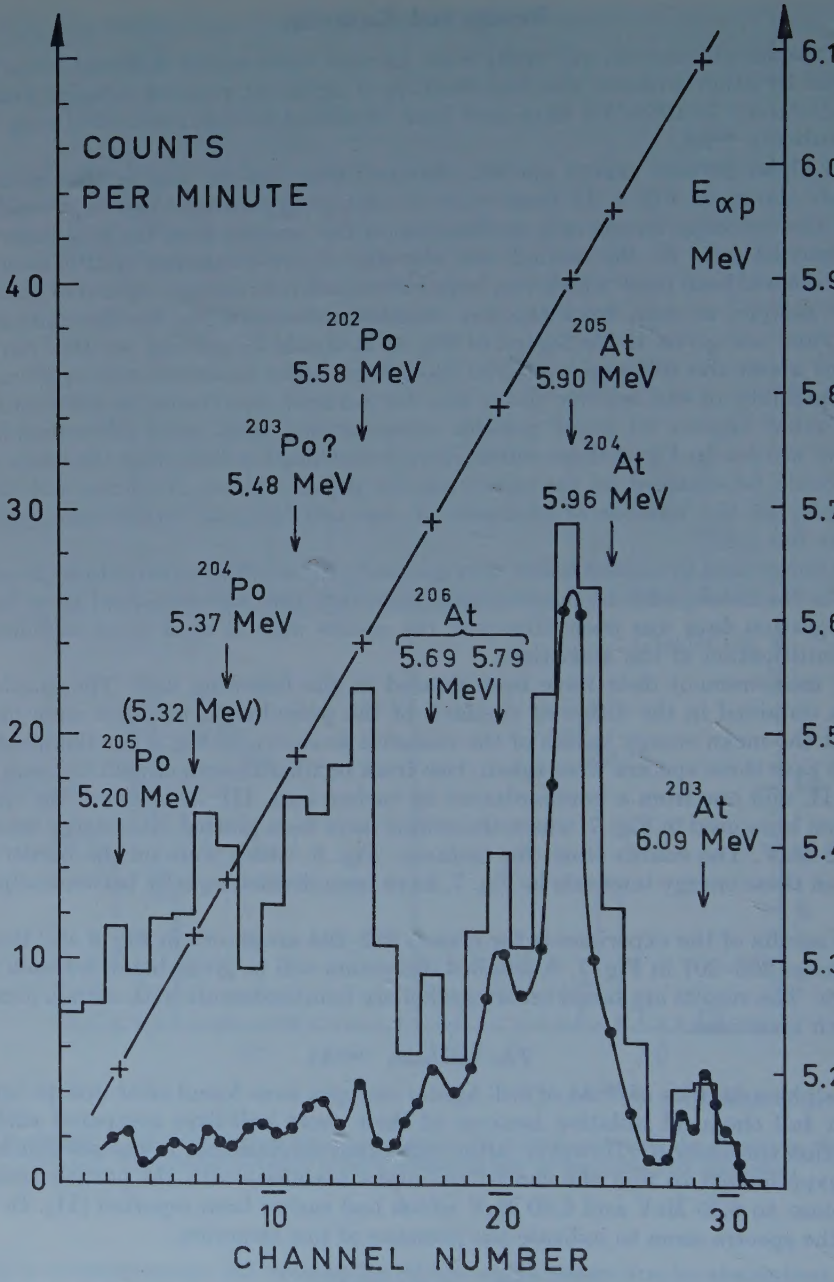

Fig. 4. Alpha-particle energy spectra of light astatine and polonium isotopes taken from about 10 and 20 minutes respectively after the end of two bombardments of gold by carbon ions. The upper spectrum, obtained from a sample which had undergone only sublimation in the cold finger crucible, shows the mean alpha activities counted in the different analyzer channels during a five minute measurement. The nuclides responsible for the activities are those indicated in the diagram.

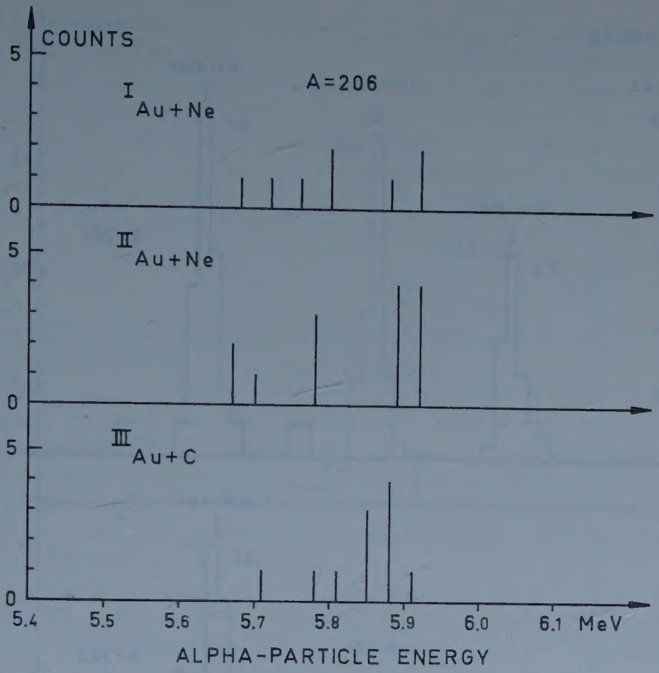

Fig. 5. Number of alpha counts obtained for mass 206 in three different experiments, I, II and III, plotted against the mean energy values of the pulse-height analyzer channels.

tion, chemical separation by extraction with DIPE and isotope separation. The result from the mass separation in one carbon ion reaction experiment is shown in Fig. 6. Thus, an alpha-particle energy of 6.13 ± 0.03 MeV was found. An indication of an alpha activity at about 6.25 MeV is also seen, which is in agreement with our results from experiments without isotope separation and also with the data reported by Hoff, Asaro and Perlman [18]. It is, however, surprising that the 6.23 MeV activity, as reported by them, was said to have a relative intensity of 36 % of the total $^{202}$At alpha activity. The low number of counts close to this higher energy in our alpha spectrum of mass 202 is not sufficient for a definite mass statement.

From the ion chamber measurements, a half-life of 3 ± 1 minutes was found, as shown in Fig. 8.

In the spectrum of mass 202 the electron capture daughter product $^{202}$Po was also found, as seen in Fig. 6. Its decay was followed in the ion chamber. The results were in good agreement with the decay data earlier reported from this institute [7, 9].

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The linear relation between the alpha-particle energies, $E_{\alpha p}$, and the channel numbers is seen from the straight line, which also gives the energy values of the individual activity peaks from the scale of the right ordinate axis.

The lower spectrum is taken from a run where the channel widths were somewhat different and for that reason the curve drawing manner has also been somewhat changed. In this run a five minute measurement was made after both sublimation and DIPE chemistry as described in the text. The activities from the polonium isotopes have here almost disappeared, but those from the astatine isotopes are left to an extent dependent upon the bombardment conditions, the chemical yields and the half-lives involved.

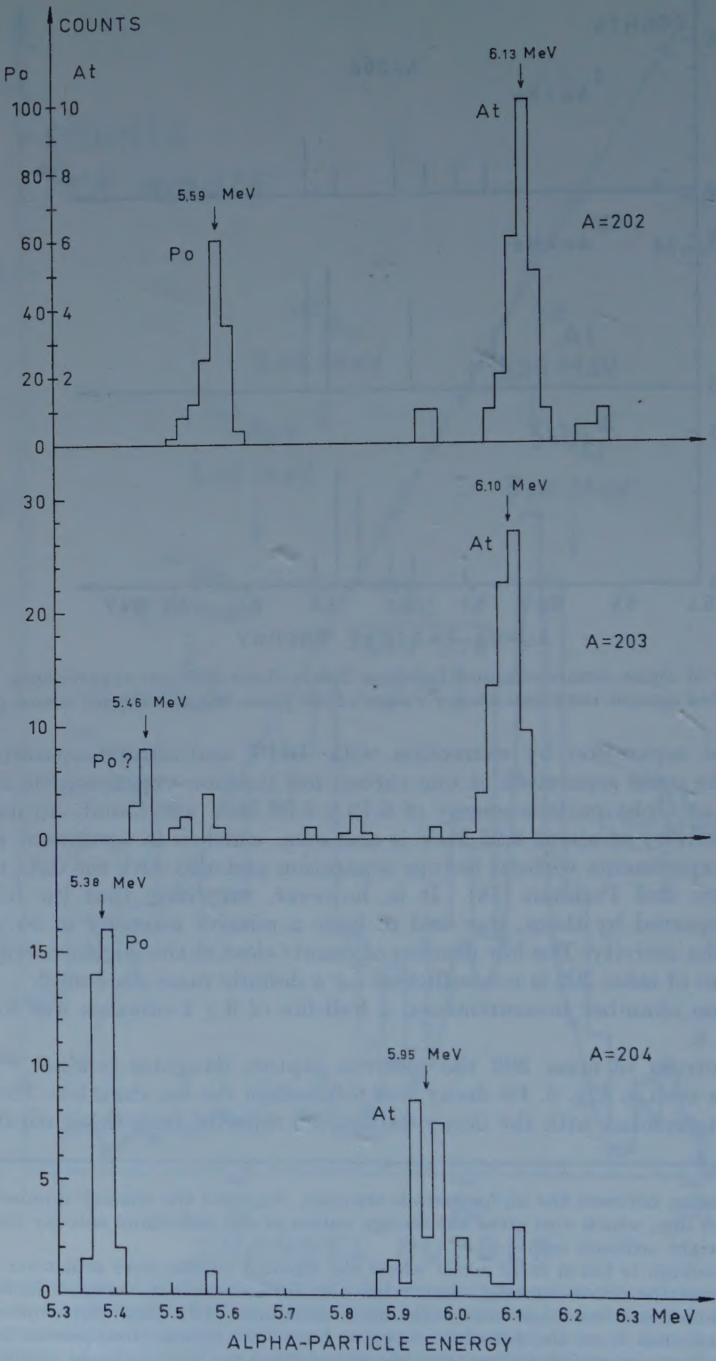

Fig. 6. Energy spectra from isotope-separated samples of the masses 202, 203, and 204.

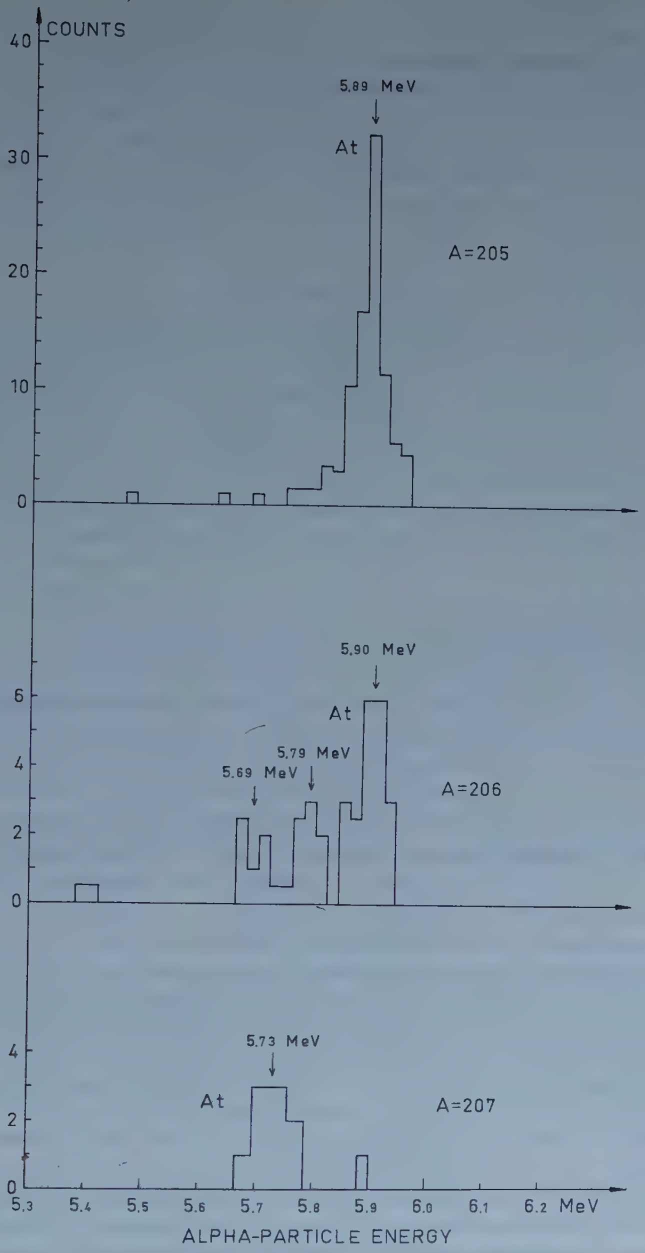

Fig. 7. Energy spectra from isotope-separated samples of the masses 205, 206, and 207.

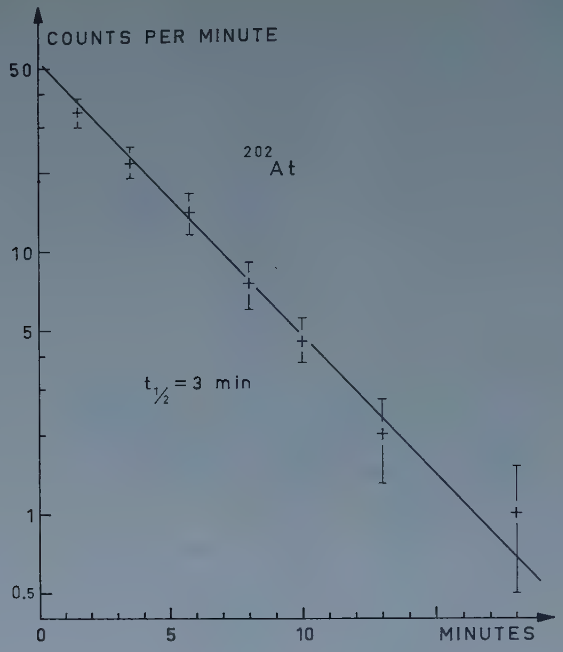

Fig. 8. Decay curve of $^{202}$At from alpha-particle pulse-height analysis.

### *The nuclide* $^{203}$At

Our results are here similar to data reported earlier for this isotope [11, 12, 14, 18] However, it was of interest to study its decay data and mass number, as it has a classification code letter D in the table of nuclides [17]. That means that the mass number has earlier not been well established.

The energy spectrum of mass 203, shown in Fig. 6, is a sum of three experiments, two bombardments of gold by carbon ions and one by neon ions. In the first of these runs the energy value obtained for the peak turned out to be somewhat higher than was expected from our other experiments. This fact, which is reflected in the spectrum, was probably partly due to a small channel drift during the time interval between the ion chamber measurements of $^{203}$At and of the standard activity $^{252}$Cf. The half-life obtained from the mass-separated samples was 7 ± 2 minutes.

In ion chamber measurements from evaporated samples which had not undergone isotope separation, the activity peak was found at the energy 6.09 ± 0.03 MeV. In several measurements this alpha-energy spectrum was resolved into three peaks. We do not think that in our measurements changes in, for instance, the channel widths, would have been responsible for the splitting of our $^{203}$At alpha peak. It should be added that the astatine sample had undergone complete chemical separation and that no alpha-decaying impurities other than the increasing daughter products were seen in the alpha spectra. In the energy spectrum for mass 203, shown in Fig. 6, the resolution was not high enough to give more than one alpha peak, even if there really were fine structure present.

There are some counts spread over a broad energy range in the spectrum. Only very few of them could be background counts not due to the sample itself. Around 5.46 MeV, there is a small activity which seems to be significant. There are good reasons to suggest that this alpha activity came from $^{203}$Po, which was reported from this institute a few years ago to have an alpha-particle energy of about 5.48 MeV and a half-life of about 45 minutes [7]. From what is known to date about the decay of $^{199}$Bi it is not probable that the activity under consideration is due to this alpha-decay daughter nuclide of $^{203}$At.

### *The nuclide* $^{204}$At

The alpha activity from $^{204}$At was studied in three bombardments of gold by carbon ions and one bombardment of gold by neon ions.

The double peaks in Fig. 6 at the energies 5.93 and 5.97 MeV had no correspondence in the individual experiments. Thus, in this case, there was no reason to believe that this splitting was due to fine structure. The mean value of 5.95 MeV for the alpha-particle energy was in close agreement with the value preliminarily reported by Hoff, Asaro and Perlman [18]. This alpha activity had not been found before 1956 [21]. It was earlier impossible to resolve this comparatively weak alpha activity from the often overwhelming activity due to $^{205}$At.

Also in the energy spectrum of mass 204 there were more counts above 6.0 MeV and below 5.92 MeV than could be explained by the background. Whether this really indicated a fine structure or was due to impurities from the adjacent masses could not be stated from our data. It seems, however, unlikely that it could have been a hydride of $^{203}$At responsible for the counts around 6.1 MeV. For more accurate results a better statistical basis must be obtained in future experiments. For similar reasons no accurate half-life determinations have been possible to date. The values obtained for the identification of the activity were in the range of $9 \pm 3$ minutes, this result being in agreement with the data given by Hoff, Asaro and Perlman [18].

The daughter product, $^{204}$Po, from electron capture decay of $^{204}$At, was found in the energy spectrum as shown in Fig. 6.

### *The nuclide* $^{205}$At

The alpha activity from $^{205}$At has been well known for many years, but it has been classified by the code symbol B [17]. The results obtained by us were in very good agreement with the earlier data [11, 12, 14, 18]. The values obtained in this study were $E_{\alpha p} = 5.90 \pm 0.03$ MeV and $t_{\frac{1}{2}} = 28 \pm 3$ minutes. From this work we can state that no doubt is left about the mass assignment. The energy spectrum is shown in Fig. 7.

### *The nuclide* $^{206}$At

No alpha activity was reported for $^{206}$At in the 1958 nuclide table [17]. An electron-capture decaying isotope of mass 206 was said to have a half-life of almost 3 hours [11, 15]. In 1959 Hoff, Asaro and Perlman [18] reported an alpha activity for $^{206}$At with a half-life of about 22 minutes and an energy of 5.699 MeV. Our study gave results similar to theirs. However, after isotope separation, we found that the alpha activity of $^{206}$At had particle energies in the range of 5.67–5.92 MeV. Even if it seems to be unlikely, the higher energies could possibly have been due to contaminations from mass 205 in the hydride form. The activities around 5.7 MeV and

5.8 MeV could, however, not at all be explained in this way. In the experiments I and II of Fig. 5 the samples had undergone cold finger sublimation before the isotope separation.

Since lead chloride was used as a standard material, any possible changes in the mass dispersion during the isotope separation should not be serious for the mass 206 compared with the lighter masses, where such possible changes had to be taken into consideration. The alpha activity was not due to impurities in the lead chloride, which was tested in a sample without astatine present.

As the results obtained by us for mass 206 were based on very few decaying atoms after the isotope separation, the half-life of this isotope could roughly be determined to be 20 ± 10 minutes. No difference in half-life could be found for the atoms decaying with the particle energy of 6.7 MeV and those with the energy of 6.9 MeV. As mentioned above, the results from the three runs, where mass 206 was studied, are shown separately in Fig. 5 and taken together in one of the spectra of Fig. 7. As there was no hope of getting considerably higher yields from further experiments in this series, we decided to present the data available even if they, in this case, must be considered as only preliminary. If, after further technical development of the method, we are able to achieve higher astatine yields, we hope to be in a position to make more definite statements about this activity.

### *The nuclide* $^{207}$At

The alpha activity of $^{207}$At is known from earlier determinations [11, 13, 14, 16, 18]. It has been classified with the code letter A [17], which means that both element and mass number are certain. Our results, presented in Fig. 7, from only one neon ion bombardment experiment, were entirely in agreement with the earlier results. $^{207}$At was not found after any carbon ion bombardment, which is due to the energetically unfavourable character of the nuclear reaction $^{197}$Au($^{12}$C, $2n$) $^{207}$At. Of this isotope, as well as of some of the lighter ones, we obtained much more activity than is seen from the diagram. However, a slight energy drift in the pulse analyzer during the automatic measurements overnight, forced us to omit the alpha activity for which no comparison with the standard activity was available. As seen from Fig. 7, the energy spectrum of mass 207 could be considered as a test of our method. In this run we got a maximum at about 5.73 MeV. A half-life of 2.0 ± 0.5 hours was found from the total 207 activity. From some rather strong samples, which had not been isotope separated we obtained $E_{\alpha p} = 5.75 \pm 0.03$ MeV and $t_{\frac{1}{2}} = 1.8 \pm 0.2$ hours.

A summary of our results in determining the alpha-decay properties and the masses of neutron-deficient astatine isotopes is shown in Table 1.

*Table 1.* Alpha-particle energies and half-lives of neutron-deficient astatine isotopes.

| Mass number | Alpha-particle energy $E_{\alpha_p}$ (in MeV) | Half-life $t_{\frac{1}{2}}$ |
|---|---|---|
| 202 | 6.13 ± 0.03; 6.25 ± 0.05? | 3 ± 1 min |
| 203 | 6.09 ± 0.03 | 7 ± 2 min |
| 204 | 5.95 ± 0.03 | 9 ± 3 min |
| 205 | 5.90 ± 0.03 | 28 ± 3 min |
| 206 | 5.70 ± 0.05; 5.80 ± 0.05; 5.90 ± 0.05? | 20 ± 10 min |
| 207 | 5.75 ± 0.03 | 1.8 ± 0.2 h |

From the neon experiments, alpha decay data for $^{208}$At and $^{209}$At were found to be in agreement with earlier reported values [11, 15]. Thus, the alpha activity found at 5.64 ± 0.03 MeV could quantitatively be separated into one 1.6 ± 0.2 hour and one 5.5 ± 0.2 hour component. In these cases no isotope separation has been made.

By combination of the alpha-decay data with those of known daughter products, it is often possible in this element region to determine branching ratios for alpha- and electron-capture decay. In several papers, where the mass assignments have not been well established, the results of such calculations have, unfortunately been of very little value. In this study we have omitted such calculations, as we hope to obtain more information about yields of daughter products, mass assignments and decay data in the near future. Also the reasons for the divergences of some of our results from those of earlier workers can then probably be more easily explained.

## ACKNOWLEDGEMENTS

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